

## HARDNESS OF NITRIDED LAYERS TREATED BY PLASMA NITRIDING

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**ABSTRACT.** Stainless steels, particularly the austenitic stainless grades are widely used in many industries due to good corrosion resistance, but very poor mechanical properties as surface hardness and wear resistance limit its possible use. Plasma nitriding is one of the few ways to increase the surface hardness of these steels, even though this will affect its corrosion resistance. This paper focuses on the description of the mechanical properties of nitrided layers in the two most widespread austenitic stainless steels AISI 304 and AISI 316L. The microstructure and properties of nitrided layers were evaluated by metallography and microhardness measurement. Surface properties of nitrided steels were characterized by Martens hardness. The results show that plasma nitriding created very hard nitrided layers with thickness about 40  $\mu\text{m}$  and microhardness about 1300  $\text{HV}_{0.05}$ . Surface hardness measurements have shown that the maximum values for both steels are about 8.5 GPa, but have different behaviour under higher loads, when the AISI 316L nitrided layer began to crack on the surface and sink.

**KEYWORDS:** Austenitic stainless steel, microhardness, plasma nitriding.

### 1. INTRODUCTION

Plasma nitriding is one of the few technologies that allow us to increase the surface hardness and abrasion resistance of austenitic steels, and thanks to this surface treatment we can use austenitic stainless steels in other more demanding applications in industries such as chemical, petrochemical food or medical articles. [1–5] The issue of nitrided layers formed on austenitic steels has been intensively solved since the 1990s, when the properties of these layers such as their depth, hardness, abrasion resistance and their influence on the corrosion resistance of steels are investigated. [6–10]. Another possible application of such treated surfaces is as a transition layer between a soft backing material such as austenitic steels and a very hard and thin PVD coating [11–14]. The aim of this paper is to describe the mechanical properties of nitrided layers formed on AISI 316L and 304 austenitic stainless steels.

### 2. EXPERIMENT

The AISI 304 steel samples were 50 x 30 mm and 2 mm thick in the unprocessed state, and the 316L steel had a 30 mm diameter and 7 mm thickness. The surface of the samples was ground and polished to a mirror finish and cleaned in an ultrasonic bath. The values of the chemical composition are given in Tab. 1. Subsequently, the samples were nitrided in plasma furnace Rübigen PN 60/60 at condition see Tab. 2. Sputter cleaning was conducted in an Ar-H<sub>2</sub> atmosphere prior to plasma nitriding with a pulsed

- DC glow discharge. All samples were cooled after plasma nitriding in vacuum.

Depth profiles of plasma nitrided layers were measured by GDOES/QDP method. GDOES measurements were performed in LECO SA-2000 equipment. Calibration of nitrogen: JK41-1N and NSC4A standards were used. Confocal laser microscope LEXT OLS 3000 was used for observation of the cross section morphology of nitrided layers. The microhardness profile was measured on the LECO AMH 47 automatic microhardness tester. The Vickers indenter was selected, the load was 0.049 N, dwell time at load was 12 s. The profile of microhardness was measured at 5 locations on each sample.

Surface hardness of nitrided samples was measured on the instrument Zwick ZHU 2.5. The Martens hardness of nitrided layers in macroscale was determined. Vickers indenter was used, load was 196 N, dwell time at load of 12 s. Average value was determined from five measurements.

### 3. RESULTS

The nitriding process was carried out under specified conditions; Methane was added to the nitriding atmosphere during the nitriding process. GDOES measurements (Figs. 1, 2) showed that the plasma nitrided layer had a different chemical composition than the layer formed during standard nitriding. In both steels there were 1 zone in the nitrided layer where the amount of carbon increased. In AISI 304 steel, this zone occurred at a depth of about 10  $\mu\text{m}$  below

		Chemical composition (wt%)						
	C	Mn	Cr	Ni	Si	P	S	Mo
AISI 304	0.045	1.78	18.7	8.75	0.48	0.030	0.001	-
AISI 316 L	0.03	1.50	16.97	10.70	0.31	0.030	0.026	2.18

TABLE 1. Chemical composition of stainless steels

	Cleaning	Nitriding
Temperature (°C)	520	550
Duration (hours)	0.5	8 h
Flow H <sub>2</sub> (l/min <sup>-1</sup> )	20	8
Flow N <sub>2</sub> (l/min <sup>-1</sup> )	2	32
Flow CH <sub>4</sub> (l/h)	0	1.5
Bias (V)	800	530
Pulse length (µs)	100	100
Pressure (Pa)	80	280

TABLE 2. Parameters of plasma nitriding process

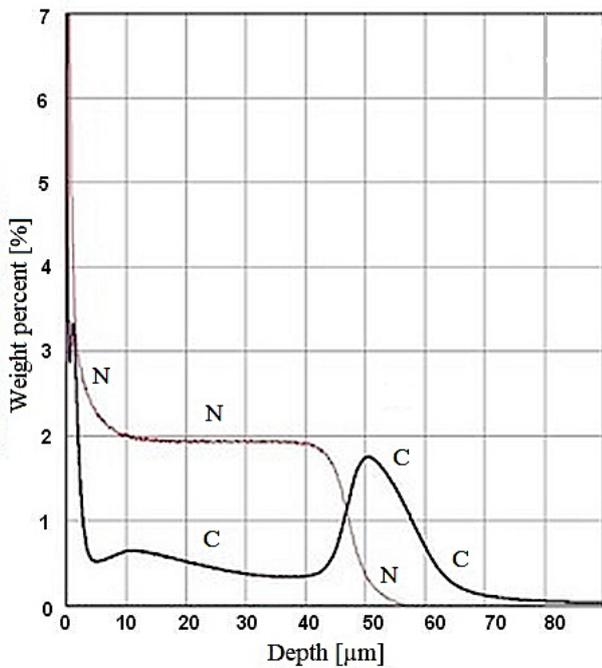


FIGURE 1. GDOES depth profile of nitrided AISI 304 steel

the surface, and this layer is very well visible in Fig. 3. The second significant zone is below the nitrided layer; this significant zone was formed during the diffusion process as carbon atoms are displaced nitrogen atoms. In AISI 316L steel, a zone of higher carbon content was also found at a depth of 15 µm below the surface, but it is not apparent in the cross-section Fig. 4. It can be clearly seen from Fig. 4 that the diffusion layer formed has a different character at the bottom, not as sharply delimited as the AISI 304 steel. The total layer thickness of the AISI 304 steel was 45 µm. In AISI 316L steel the nitrided layer reached a thickness of 40 µm.

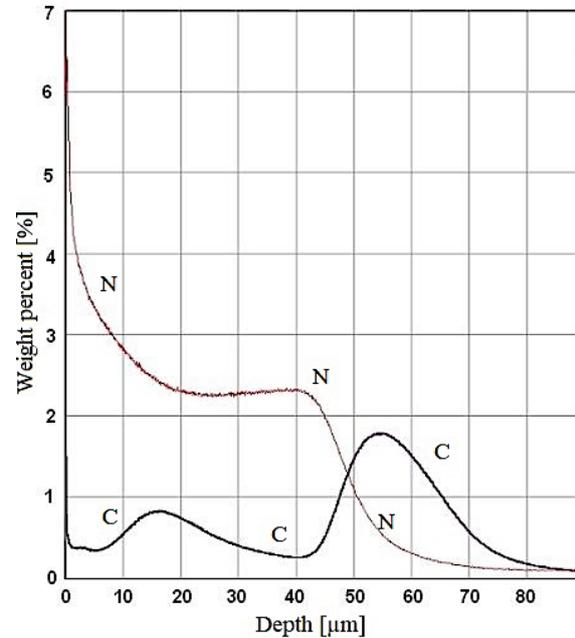


FIGURE 2. GDOES depth profile of nitrided AISI 316L steel

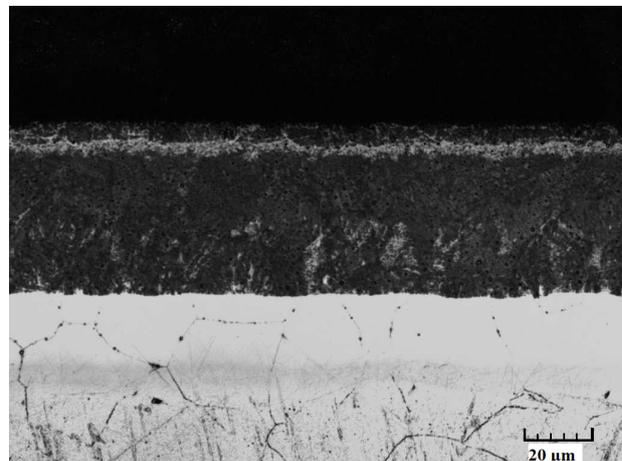


FIGURE 3. Micrographs of the nitrided layer of the AISI 304 steel

The thickness and profile of the nitrided layers were measured by microhardness see Fig. 5., 6., thickness of both nitrided layers of AISI 304 and AISI 316L were in good agreement with cross section morphology. The highest values of microhardness were similar but for AISI 316L was higher, reached 1383 HV<sub>0.05</sub> and for AISI 304 the highest values of microhardness reached 1200 HV<sub>0.05</sub>.

The highest values of surface hardness were the

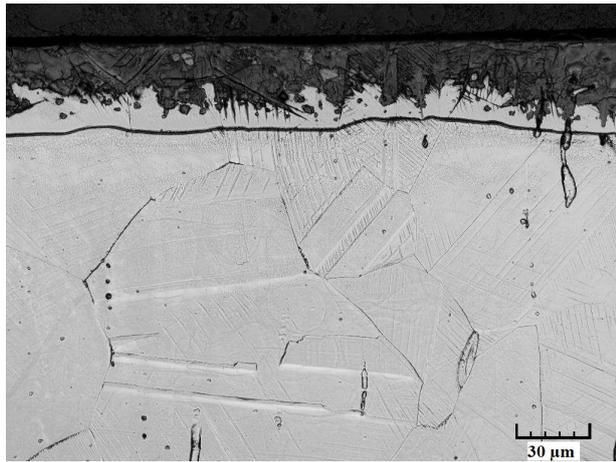


FIGURE 4. Micrographs of the nitrided layer of the AISI 316L steel

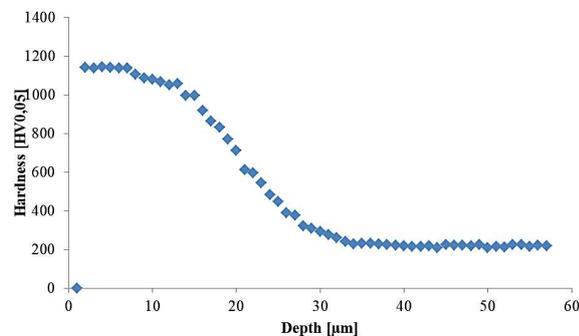


FIGURE 5. Microhardness profile of nitrided AISI 304 steel

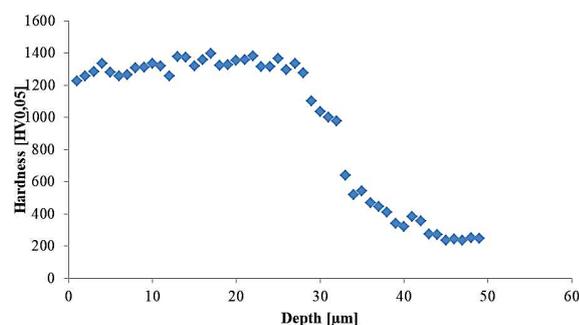


FIGURE 6. Microhardness profile of nitrided AISI 316L steel

same for both of the steels reached 8.5 GPa. As can be seen from the graphs Fig. 7. 8., each nitrided layer behaved differently during loading. With the AISI 304 material, the curve to an indentation depth of about 6 micrometres was stable and then gradually decreased with increasing indentation depth. Thus, in the case of AISI 316L, the curve was diametrically different; the layer exhibited stability to an indentation depth of about 15  $\mu\text{m}$  and then ruptured and continued to crack even at an increasing indentation depth.

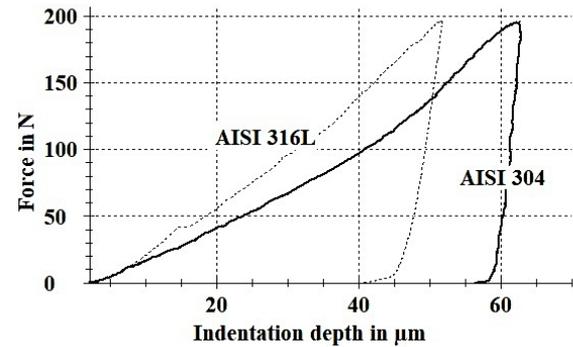


FIGURE 7. Force - displacement curve of nitrided layers

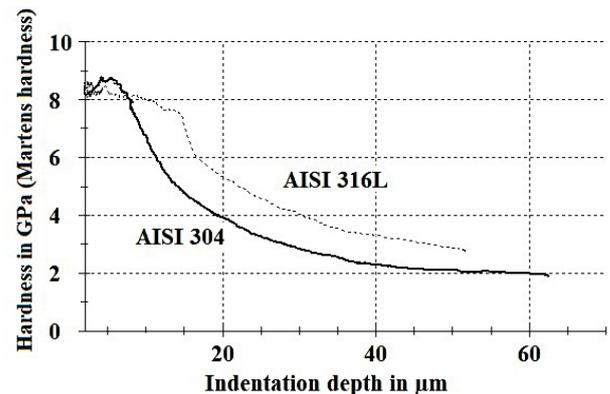


FIGURE 8. Hardness vs indentation depth of nitrided layers

#### 4. CONCLUSIONS

The results of the experiment showed that plasma nitriding creates a sufficiently deep nitrided layer on both steels that can be used as a transition layer between a soft substrate and a PVD coating. In AISI 316L steel, the nitrided layer has more suitable properties because it achieves higher hardness and thickness of nitrided layer than AISI 304 steel nitrided layer which has lower microhardness and thickness and in addition, at higher loads, the surface of the nitrided layer is broken.

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